

## ANALYSIS CASE NARRATIVE

### General Information

61 water samples were received 3/25 – 3/29/19 for per- and polyfluoroalkyl substances (PFAS) analysis. All holding times were met. The designated analyst for these samples was Larry Zintek; however, the final data package and report was completed by Danielle Kleinmaier. Any questions concerning this data should be directed to Larry Zintek, who can be reached at (312) 886-2925.

### Sample Analysis and Results

Sample preparation and analysis occurred via the Chicago Regional Laboratory standard operating procedure (CRL SOP) OM021 Ver. 2 with the deviation outlined in pen-and-ink change #11017 (revised surrogate list). Currently, target analyte 6:2 FTS and its labelled surrogate are not reported by this SOP due to repeated QC failures (pen-and-ink change #15481), but since the client specifically requested it, 6:2 FTS and its labelled surrogate will appear in this report qualified as 'Research'.

All of the field sample data in work orders (WOs) 1903009, 10, and 11 has been qualified estimated ('J') due to being sub-sampled from the 250 mL high-density polyethylene (HDPE) sample bottles provided to the CRL.

The data reported herein meets the requirements referenced in the analytical SOP and any laboratory specifications stated in the document "[PLACEHOLDER]" with the exceptions noted in the Quality Control section below. Some sample concentrations may have changed between the preliminary and final report. See the report for detailed sample results.

### Quality Control

All quality control audits not mentioned below were within the limits specified in the CRL SOP or did not result in qualification of the data. See the report for detailed QC results.

Several matrix QC failures were observed in these data sets (i.e. surrogate recovery failures and matrix spike recovery failures). As these field samples are comprised of sea water, this is most likely due to the salt content of the samples, which is adversely affecting the recovery of the PFAS analytes.

### WO 1903009

#### *Second Source Verification*

6:2 FTS in the second source verification standard analyzed during both the undiluted and diluted sequences of batch B19C023 had a percent difference (%D) between the calculated concentration and the expected concentration of 180% and 176%, respectively, failing the SOP %D criteria of  $\pm 30\%$ . Affected data for 6:2 FTS are qualified appropriately.

#### *Surrogate Recovery*

Surrogate M2PFTreA was recovered below the SOP control limits in 1903009-01, -02, -04 through -14, -16, B19C023-DUP1, -MS1, and -MSD1. Surrogate MPFD<sub>o</sub>A was recovered below the limits in 1903009-10, -13, and B19C023-MS1. Surrogate M6:2 FTS was recovered above the limits in 1903009-03 through -10, -13, -14, -16, B19C023-DUP1, -MS1, and -MSD1. Surrogate MNEtFOSAA was recovered below

the limits in 1903009-10, -13, -14, and B19C023-DUP1. Surrogate MNMeFOSAA was recovered below the limits in 1903009-10 and -13. All affected data are qualified appropriately.

#### *Reporting Limit (RL) Check Recovery*

PFOS in B19C023-MRL2 and 6:2 FTS in B19C023-MRL1 and -MRL2 were recovered above the SOP control limits. Affected data for PFOS and 6:2 FTS are qualified appropriately.

#### *Matrix Spike Recovery*

PFPeA, PFOA, and PFDA were recovered below the SOP control limits in B19C023-MS1, PFHxS was recovered below the limits in B19C023-MSD1, and PFHpA, PFNA, PFUnA, PFDoA, PFTriA, PFTreA, 6:2 FTS, N-MeFOSAA, and N-EtFOSAA were recovered below the limits in both B19C023-MS1 and -MSD1. Affected data in the native sample (1903009-16) are qualified appropriately.

#### *Qualitative Identification*

For any analyte, when the ion ratio limit of  $\pm 30\%$  is exceeded, the affected data are qualified 'I'.

#### WO 1903010

##### *Initial Calibration*

The coefficient of determination for the initial calibration of PFTreA and M2PFTreA in the undiluted sequence of batch B19C027 was 0.98, failing the SOP criteria of  $\geq 0.99$ . Affected data for PFTreA and M2PFTreA are qualified appropriately.

##### *Second Source Verification*

6:2 FTS in the second source verification standard analyzed during the undiluted and diluted sequences of batch B19C027 had a %D between the calculated concentration and the expected concentration of 170%, 162%, and 172%, respectively, failing the SOP %D criteria of  $\pm 30\%$ . Affected data for 6:2 FTS are qualified appropriately.

##### *Surrogate Recovery*

Surrogate M9PFNA was recovered below the SOP control limits in B19C027-MS1. Surrogate M2PFTreA was recovered below the limits in 1903010-01 through -09, B19C027-DUP1, -MS1, and -MSD1. Surrogate M7PFUnA was recovered below the limits in 1903010-06, -08, -09, B19C027-DUP1, and -MS1. Surrogate MPFDoA was recovered below the limits in 1903010-04 through -09, B19C027-DUP1, -MS1, and -MSD1. Surrogate M6:2 FTS was recovered above the limits in 1903010-02, -03, and B19C027-MSD1. Surrogate MNEtFOSAA was recovered below the limits in 1903010-06 through -09, B19C027-DUP1, -MS1, and -MSD1. Surrogate MNMeFOSAA was recovered below the limits in 1903010-06, -08, -09, B19C027-DUP1, -MS1, and -MSD1. All affected data are qualified appropriately.

##### *RL Check Recovery*

6:2 FTS in B19C027-MRL2 was recovered above the SOP control limits. Affected data for 6:2 FTS are qualified appropriately.

##### *Matrix Spike Recovery*

PFDA and PFOS were recovered below the SOP control limits in B19C027-MS1 and PFNA, PFUnA, PFDoA, PFTriA, PFTreA, N-MeFOSAA, and N-EtFOSAA were recovered below the limits in both B19C027-MS1 and -MSD1. Affected data in the native sample (1903010-07) are qualified appropriately.

##### *Qualitative Identification*

For any analyte, when the ion ratio limit of  $\pm 30\%$  is exceeded, the affected data are qualified 'I'.

WO 1903011*Second Source Verification*

6:2 FTS in the second source verification standard analyzed during both the undiluted and diluted sequences of batch B19C028 had a %D between the calculated concentration and the expected concentration of 162% and 172%, respectively, failing the SOP %D criteria of  $\pm 30\%$ . Affected data for 6:2 FTS are qualified appropriately.

*Surrogate Recovery*

Surrogate M8PFOA was recovered below the SOP control limits in 1903011-07 and -09. Surrogate M9PFNA was recovered below the limits in 1903011-05 through -07, -09, and -10. Surrogate M6PFDA was recovered below the limits in 1903011-06, -07, -10, and -11. Surrogate M4PFHpA was recovered below the limits in 1903011-06 and -07. Surrogate M2PFTreA was recovered below the limits in 1903011-01 through -12, B19C028-DUP1, -MS1, and -MSD1. Surrogate M7PFUnA was recovered below the limits in 1903011-05 through -11. Surrogates MPFDoA and MNEtFOSAA were recovered below the limits in 1903011-04 through -11, B19C028-DUP1, and -MSD1. Surrogate M8:2 FTS was recovered below the limits in 1903011-12. Surrogate M6:2 FTS was recovered above the limits in 1903011-01 through -04, -06, B19C028-MS1, and -MSD1. Surrogate MNMeFOSAA was recovered below the limits in 1903011-03 through -11 and B19C028-DUP1. All affected data are qualified appropriately.

*RL Check Recovery*

6:2 FTS was not recovered in either B19C028-MRL1 or -MRL2. As a result, the RL for 6:2 FTS was raised to 20 ng/L in batch B19C028.

*Blank Spike Recovery*

6:2 FTS was recovered below the SOP control limits in B19C028-BS1. However, 6:2 FTS was recovered within the limits in B19C028-BSD1. Therefore, no data was affected.

*Matrix Spike Recovery*

PFHxS, FOSA, and PFDS were recovered below the SOP control limits in B19C028-MS1 and PFNA, PFDA, PFUnA, PFDoA, PFTriA, PFTreA, N-MeFOSAA, and N-EtFOSAA were recovered below the limits in both B19C028-MS1 and -MSD1. Affected data in the native sample (1903011-11) are qualified appropriately.

*Qualitative Identification*

For any analyte, when the ion ratio limit of  $\pm 30\%$  is exceeded, the affected data are qualified 'I'.

WO 1903012*Second Source Verification*

6:2 FTS in the second source verification standard analyzed during both the undiluted and diluted sequences of batch B19C030 had a %D between the calculated concentration and the expected concentration of 172% and 167%, respectively, failing the SOP %D criteria of  $\pm 30\%$ . PFOS in the second source verification standard analyzed during the diluted sequence of batch B19C030 had a %D between the calculated concentration and the expected concentration of -31.1%, also failing the SOP %D criteria. Affected data for 6:2 FTS and PFOS are qualified appropriately.

*Surrogate Recovery*

Surrogate M9PFNA was recovered below the SOP control limits in 1903012-07 and -11. Surrogate M6PFDA was recovered below the limits in 1903012-05 through -11, B19C030-DUP1, -MS1, and -MSD1. Surrogate M2PFTreA was recovered below the limits in 1903012-01 through -11, B19C030-DUP1, -MS1, and -MSD1. Surrogate M7PFUnA was recovered below the limits in 1903012-04 through -11, B19C030-DUP1, -MS1, and -MSD1. Surrogate MPFDoA was recovered below the limits in 1903012-02 through -11, B19C030-DUP1, -MS1, and -MSD1. Surrogate M8PFOS was recovered below the limits in 1903012-05, -08, and -11. Surrogate M6:2 FTS was recovered above the limits in 1903012-01 through -11, B19C030-DUP1, -MS1, and -MSD1. Surrogates MNEtFOSAA and MNMeFOSAA were recovered below the limits in 1903012-03 through -11, B19C030-DUP1, -MS1, and -MSD1. All affected data are qualified appropriately.

#### *RL Check Recovery*

6:2 FTS in B19C030-MRL1 and -MRL2 was recovered above the SOP control limits. Affected data for 6:2 FTS are qualified appropriately.

#### *Matrix Spike Recovery*

PFOA, PFOS, FOSA, and PFNS were recovered below the SOP control limits in B19C030-MS1, 6:2 FTS was recovered above the limits in B19C030-MSD1, and PFNA, PFDA, PFUnA, PFDoA, PFTriA, PFTreA, N-MeFOSAA, N-EtFOSAA, and PFDS were recovered below the limits in both B19C030-MS1 and -MSD1. The relative percent difference (RPD) between B19C030-MS1 and -MSD1 for PFDoA, PFTriA, PFTreA, and PFDS was also above the SOP limit. Affected data in the native sample (1903012-10) are qualified appropriately.

#### WO 1903013

#### *Initial Calibration*

The two lowest initial calibration standard points for PFOS in the undiluted sequence of batch B19D001 had a %D between the calculated concentration and the expected concentration of 56.5% and 72.1%, respectively, failing the SOP %D criteria of  $\pm 30\%$ . The two points were excluded from the initial calibration curve for PFOS and the RL was raised to 40 ng/L in batch B19D001.

#### *Second Source Verification*

6:2 FTS in the second source verification standard analyzed during the undiluted and diluted sequences of batch B19D001 had a %D between the calculated concentration and the expected concentration of 172%, 167%, and 225%, respectively, failing the SOP %D criteria of  $\pm 30\%$ . PFOS in the second source verification standard analyzed during the diluted sequence of batch B19D001 had a %D between the calculated concentration and the expected concentration of -31.1%, also failing the SOP %D criteria. Affected data for 6:2 FTS and PFOS are qualified appropriately.

#### *Surrogate Recovery*

Surrogate M2PFTreA was recovered below the SOP control limits in 1903013-01 through -09, -11, -12, B19D001-DUP1, -MS1, and -MSD1. Surrogate M7PFUnA was recovered below the limits in 1903013-06 and -07. Surrogate MPFDoA was recovered below the limits in 1903013-01 through -04, -06 through -08, -11, -12, B19D001-DUP1, -MS1, and -MSD1. Surrogate M6:2 FTS was recovered above the limits in 1903013-01 through -09, -11, B19D001-DUP1, -MS1, and -MSD1. Surrogate MNEtFOSAA was recovered below the limits in 1903013-02 through -04, -06 through -08, B19D001-DUP1, -MS1, and -MSD1. Surrogate MNMeFOSAA was recovered below the limits in 1903013-04, -06 through -08, B19D001-DUP1, and -MSD1. All affected data are qualified appropriately.

*RL Check Recovery*

6:2 FTS in B19D001-MRL1 and -MRL2 was recovered above the SOP control limits and PFOS was not recovered in either B19D001-MRL1 or -MRL2. Affected data for 6:2 FTS are qualified appropriately. The RL for PFOS was already raised to 40 ng/L in batch B19D001.

*Matrix Spike Recovery*

PFNA, PFUnA, PFDoA, PFTriA, PFTreA, PFOS, N-MeFOSAA, N-EtFOSAA, and PFDS were recovered below the SOP control limits in both B19D001-MS1 and -MSD1. Affected data in the native sample (1903013-09) are qualified appropriately.